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GRINDING KINETICS AND EQUILIBRIUM STATES

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16. Abstract  The temporary and permanent equilibrium occurring during the initial stage of cement grinding does not indicate the end of comminution, but rather an increased energy consumption during grinding. The constant dynamic equilibrium occurs after a long grinding period indicating the end of comminution for a given particle size. Grinding equilibrium curves can be constructed to show the stages of comminution and agglomeration for certain particle sizes.			
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## GRINDING KINETICS AND EQUILIBRIUM STATES

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### Introduction

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The particle size distribution provides most of the information at a given time about the physical state of a ground product. Domestic experience of many years standing indicates that the Rosin-Rammler-Sperling-Bennett (RRSB) equation can be used with good success for the characterization of the particle-size distribution of ground materials in the silicate industry (quartz, limestone, cement, etc.) [1]. Study of the grinding process on the basis of the RRSB equation supports unequivocally the static viewpoint, however, it can be used only indirectly to follow the time-dependent changes by means of static parameters, such as the uniformity factor ( $n$ ) or the fineness ( $\bar{x}$ ).

Many attempts have been made to derive a time-dependent function for a set particle size [2, 3, 4, etc.]; however, no generally valid correlation similar to RRSB has been found; in every case, the validity was limited to the Rittinger segment.

In the actual practice in the silicate industry the Tovarov is used most often to describe the time-dependent course of the grinding process.

A previous study of the validity of the Tovarov equation revealed that it is not applicable to all the materials that we have investigated; it was also found that as far as the various particle-size ranges of a given material are concerned, the equation becomes valid for different grinding period ranges. The segments concerning the individual particle sizes corresponding to the Tovarov equation do not appear necessarily at the beginning of the grinding process (i.e., during the period of rough grinding).

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\*Numbers in the margin indicate pagination in the foreign text.

From these results we concluded that the changes of the sieve residues which indicate the time-related course of the grinding, must be studied in greater detail, including also the period of fine grinding.

### Experimental Materials and Methods

Natural minerals (talc, gypsum, limestone, calcite, aplite and quartz) and products of the cement and limestone industries (burnt lime, cement clinkers) were used as experimental materials. We selected four materials - calcite, quartz and two kinds of cement clinkers to illustrate the obtained results. The most important characteristics of the studied materials are summarized in Table 1.

The laboratory grinding tool used for our experiments consisted of a 5-liter capacity, 20 x 20 mm mill, operated at 68 rpm, weighing 8 kg and provided with a steel-filled steel mantle. The materials were prepared for grinding in a hammer crusher (upper particle-size limit of 3.4 mm). A batch size of 1 kg was used in the experimental mill. We continued the milling for at least 5 hours. Samples were taken by emptying the mill completely at set time periods.

Up to a size of 60  $\mu$  we determined the particle-size distribution of the ground products by sieving, and below that limit, by sedimentation in a Robinson-Kohn device. We used absolute alcohol, respectively distilled water as dispersing agent, and calcium chloride, respectively sodium pyrophosphate as stabilizer. The obtained results are summarized in Table 2.

### Types of Curves Characterizing the Time-Dependent Changes of the Sieve Residue and the Grinding Rate

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The grinding kinetics were analyzed on the basis of Huttig's work [5]. From his experiments with a large number of materials, Huttig assumed that there is an "equilibrium state of grinding", however without defining the concept.

In our studies concerning the changes of the sieve residue as a function of the grinding time,  $(R(t))$ , we started from the assumption that the product of the grinding process is the resultant of a comminution (decrease of the sieve residue) and a roughening process (increase of the sieve residue due to the interaction between the particles).

In view of the above:

we observe improved fineness, if at a given moment the comminution is larger than the interaction of the particles;

we observe increased roughening if at a given moment the comminution is less than the particle interaction;

if at a given moment the comminution and the particle interaction are identical, a dynamic equilibrium is established. Such an equilibrium may occur with a transient character at the start of the initial period of the grinding process, or in general with an enduring character in a steady-state range for extended grinding periods.

We examined also the time-dependent change of the grinding rate for certain given materials. The grinding rate is defined as the first differential quotient according to time of the fall-over function:

$$\frac{\partial D(x, t)}{\partial t} = -\frac{\partial R(x, t)}{\partial t} \quad (1)$$

According to this concept the grinding rate is a characteristic suited to define the quantity of material, the size of the particles which pass through the size  $x$  within a unit time from either direction for a given grinding period  $t$ ; if the fineness is enhanced, this characteristic is positive; in case of increased roughening it is negative, and if there is no change, it is zero:

if  $\frac{\partial D}{\partial t} > 0$  , the sieve residue decreases with time. In that case the comminution is stronger than the particle interaction;

if  $\frac{\partial D}{\partial t} < 0$  , the sieve residue increases as a function of time. the comminution process is less marked than the interaction of the particles;

if  $\frac{\partial D}{\partial t} = 0$  , the sieve residue remains unchanged for a given grinding time. This indicates the establishment of the dynamic equilibrium state. /443

The  $\frac{\partial D}{\partial t} = 0$  equality at size  $x$  and grinding time  $t$  can be established in several ways:

$\frac{\partial D(x, \tau_0)}{\partial t} = 0$  and  $\frac{\partial D(x, t)}{\partial t}$  functions change signs from the positive into the negative direction at  $t = \tau_0^*$  point of time. At that time function  $D(x, t)$  has a maximum and function  $R(x, t)$  a minimum. A transient dynamic equilibrium state is established at time  $\tau_0$ , whereby the trend toward enhanced fineness changes into increased roughness.

$\frac{\partial D(x, \tau_0)}{\partial t} = 0$  and  $\frac{\partial D(x, t)}{\partial t}$  functions change sign from the positive direction at a time  $t = \tau_0$ . At that time  $D(x, t)$  has a minimum and  $R(x, t)$  a minimum. This is also a transient dynamic equilibrium, whereby at time  $\tau_0$  the increasing roughness changes into increasing fineness;

$\frac{\partial D(x, \tau_0)}{\partial t} = 0$  and  $\frac{\partial D(x, t)}{\partial t} > 0$  before and after time  $t = \tau_0$ . At that time  $D(x, t)$  and  $R(x, t)$  have a horizontal tangent at the time  $\tau_0$ . This is also a transient dynamic equilibrium state, similarly manifested by the decrease of the grinding rate to zero, although the process fundamentally tends toward enhanced fineness; /444

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\* $\tau$  designates the grinding time creating the equilibrium state

$$\frac{\partial D(x,t)}{\partial t} = 0 \quad \text{in the } \tau_1 \leq t \leq \tau_2 \text{ grinding time range.}$$

This signifies that in the  $\tau_1 \leq t \leq \tau_2$  grinding time range  $D(x,t) = \text{constant}$  and  $R(x,t) = \text{constant}$ . This is a dynamic equilibrium with a steady state character, the establishment of which indicates the completion of the trend toward enhanced fineness with respect to the given particle size.

Combining the time-related curves of all experimental materials with respect to the sieve residue, respectively the grinding rate, we obtain two kinds of  $R(t)$ , respectively  $D_x(t)$  curves (Figure 1).

Within these two types of curves we can distinguish four segments:

First segment (A - B): the segment with an undulating slope. Its basic trend is toward enhanced fineness. The two types of curves diverge from each other in the course of this segment. Type I curve contains alternately minima and maxima, while Type II curve contains only inflection points. They include also inflection points with a horizontal tangent.

We interpreted these differences on the basis of the evolution of the rate curves ( $D_x(t)$ ).

In the case of Type I rate curve, the range of negative values of the grinding rate indicates increasing roughening, the zero position signifies the transient dynamic equilibrium, while the range of positive values refers to improving fineness. The zero locations within the undulating segment of the curve (that is, locations of the maxima or minima of the sieve-residue function) indicate the establishment of a transient dynamic equilibrium state which is a consequence of the particle interaction. In this case the particle interaction causes an increase of the sieve residue. The establishment of the dynamic equilibrium state in this segment does not imply the termination of the trend toward increasing fineness.



Only Type II rate curve the undulation of the A - B segment occurs only between positive values. In this case the particle

interaction slows down the decrease of the sieve residue, respectively causes the undulating character within positive values (decrease, respectively increase) of the grinding rate.

Second segment (B - C): smoothly sloping segment, characteristic of the trend toward enhanced fineness. It can be described by the Tovarov equation. In this segment the grinding rate decreases continuously.

Third segment (C - D): the sloping segment is followed by a dynamic equilibrium segment of a steady-state character.

The zero value of grinding rate appearing here indicates the termination of the trend toward increasing fineness with respects to size  $x$ , even if the zero-rate range as a function of time exhibits only a transient character, i.e., it shrinks into a point.

Fourth segment (D - E): an unchanged  $R(t)$  segment is followed by a rising one; it is the segment, in which a steady particle interaction, respectively increased roughening take place. This is confirmed unequivocally by the increased sieve residue and the negative values of the grinding rate.

It should be pointed out that all of the examined one-component minerals follow Curve II, while most of the cement clinkers follow Curve I.

## Sieve-Residue and Grinding-Rate Surfaces

The static processing mode of the sieve residue investigation provides information about the overall grinding product for a set grinding period, and the dynamic processing for the overall grinding time range for individual sizes. However, the grinding process does not take place according to individual particle sizes and set grinding periods but involves all of these factors.

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In the following we shall consider the sieve residue values as a two-variable surface depending on the selected grinding time and the studied sieve dimensions. Study of the sieve residue at fixed grinding times for a given series of sieve dimensions (static processing), respectively at fixed sieve dimensions with given grinding times (dynamic processing) yield identical results which can be organized according to two variables.

The sieve-residue surfaces of the selected samples are shown in Figures 2 to 5. The sections parallel with the axes of the sieve-residue surface  $R(x,t)$  contain at fixed grinding times ( $t = \text{constant}$ ), the  $R_t(x)$  sieve-residue particle size; at a fixed particle size ( $x = \text{constant}$ ) they contain the sieve-residue grinding-time curves  $R_x(t)$  (Figures 2/1 - 5/1).

The two-variable  $D(x,t)$  surface describes the grinding rate that we have derived previously from the two-variable sieve-residue function, in the overall sieve-size and grinding-time range.

The grinding-rate surfaces of the selected samples are shown in Figures 2/2 - 5/2. The sections of the surface parallel with the time axis indicate the particle size ( $x = \text{constant}$ ; they are the previously presented  $D_x(t)$  grinding rate - time curves), while the sections of the grinding surface parallel with the size axis set the grinding time ( $t = \text{constant}$ ,  $D_t(x)$  are rate-particle size curves).

We are also presenting the  $D_t(x)$  curves separately on Figures 6 to 8. The grinding rate - particle size curves exhibit a bell-shape character; they have a maximum, and in some cases (for example, in the case of calcite) also a secondary maximum. When the grinding time become longer, the curves become increasingly flatter; for some materials they even enter into the range of negative values. As the curves become increasingly flatter, the maximum shifts toward the decreasing sizes. The  $D_t(x)$  curves can be used also for the quantitative evaluation of the time-dependent course of the grinding process.

The grinding-rate surface provides an opportunity to generalize the concept of the grinding equilibrium state defined for an individual particle size.

#### The Equilibrium-State Curve of Grinding

The dynamic equilibrium state with reference to a given size may appear at the zero value of the grinding rate ( $D_t(x) = 0$ ) which defines geometrically the intersection of the grinding rate - time curve and the time axis. In case of a given size, the dynamic equilibrium state can have a transient or a steady-state character; it may be applicable for a given point or points of time, or for a certain duration, but in any case it separates individual segments of the trend toward increased fineness or roughness from each other.

The location of the dynamic equilibrium state with reference to individual sizes is contained on the rate surface by the plane curve satisfying the condition  $D(x,t) = 0$  which is geometrically the line of intersection of the surface and the grinding time - particle size base plane. On the surface, this curve is the boundary line of the trends toward increased fineness and roughness, the "trajectory" of the dynamic equilibrium state evolved in the course of the grinding, i.e., the equilibrium state diagram of grinding (Figures 2/2 - 5/2).

In agreement with the character of the dynamic equilibrium, the state curve may appear during the initial segment of the grinding; in that case it delimits the area of transient "coalescence" due to the particle interaction, or it may delimit the "coalescence" area which became steady as the grinding progresses, i.e., it indicates the termination of the grinding process.

The state curves of the selected materials are shown in the figure of the grinding rate surfaces.

In the case of calcite and quartz, as the grinding time progresses a single state curve from the large toward the smaller particle sizes appears, separating the steady-state "coalescence" area from the range of increasingly finer sizes. The difference between the two lies in the fact that in case of calcite the equilibrium curve evolves after relatively short grinding times (Figure 2/2), while in the case of quartz it is formed only after longer grinding periods. The gradation indicates the appearance of the constant equilibrium segments (Figure 3/2).

In the case of clinker specimens, the evolution of an equilibrium curve delimiting the transient coalescence is observed; the evolution of the equilibrium curve delimiting the steady-state coalescence starts at Sample 2 at long grinding periods from small particle sizes. (Figures 4/2 - 5/2).

### Important Findings

In addition to the static mode of processing the sieve-residue values, we applied also the dynamic method for the evaluation of the grinding process. This led to the determination of the general shape of the  $R_x(t)$  time functions with respect to the given particle size.

The time-dependent changes of the  $R_x(t)$  sieve residue and  $D_x(t)$  grinding rate pertaining to a given particle size can be

characterized by two types of curves which differ in their evolution of the initial portion of the grinding.

In the case of materials ground according to Type I curve, the particle interaction induces in a transient manner the increase of the sieve residue, respectively the change of the grinding rate from the positive to the negative direction. /447

In materials which are ground according to Type II curve, the particle interaction simply slows down the decrease of the sieve residue, respectively causes the undulating change of the grinding rate between positive values (decrease, respectively increase).

On the basis of the analysis of the  $R_x(t)$  sieve-residue, respectively  $D_x(t)$  rate time curves belonging to a given particle size, we conclude that as a result of the particle interaction a transient steady-state dynamic equilibrium may be established in the course of the grinding; this is manifested in the constancy of the sieve residue and the decrease of the grinding rate to zero.

The transient dynamic equilibrium which is established at the start of the grinding, brings on an increase of the grinding energy consumption but it does not signify the termination of the trend toward enhanced fineness. The establishment of the transient dynamic equilibrium is a consequence of a weak coalescence, the aggregation of the particles. During the continued grinding the coalesced particles are again comminuted; the basic trend of the process is toward the decrease of the particle size, i.e., enhanced fineness.

The steady-state dynamic equilibrium state appears generally when the material is ground for a long time and it signifies completion of the comminution for a given particle size. Apparently, from this point on the grinding energy is used only to maintain the equilibrium state, but actually it induces the superficial, respectively mechanochemical activation of the particles. When the /448

grinding is continued the coalesced particles are not comminuted again; the increase of the particle size becomes the determining process. It follows from the nature of the process (increase of the sieve residue and negative values of the grinding rate) that such a state is created as a result of the appearance of stronger interactions, the agglomeration.

In the course of the grinding the equilibrium state is established for different particle sizes at different grinding times.

By combining the grinding time according to a two-variable surface of the grinding rate (size and grinding time), the static and dynamic processing provided an opportunity to illustrate the grinding process direction and to plot the  $D_t(x)$  grinding rate - particle size curve that can be used to evaluate quantitatively the progress of the grinding process.

With the help of the two dimensional  $D(x,t)$  we interpreted the grinding equilibrium state curve which contains the particle size - grinding time locations of the equilibrium states at specific particle sizes. The grinding equilibrium state curve delimits in every case the comminution and roughening, respectively their various segments.

Analysis of the grinding kinetics confirmed our earlier findings concerning the existence of qualitatively different gradations of the particle interaction [6].

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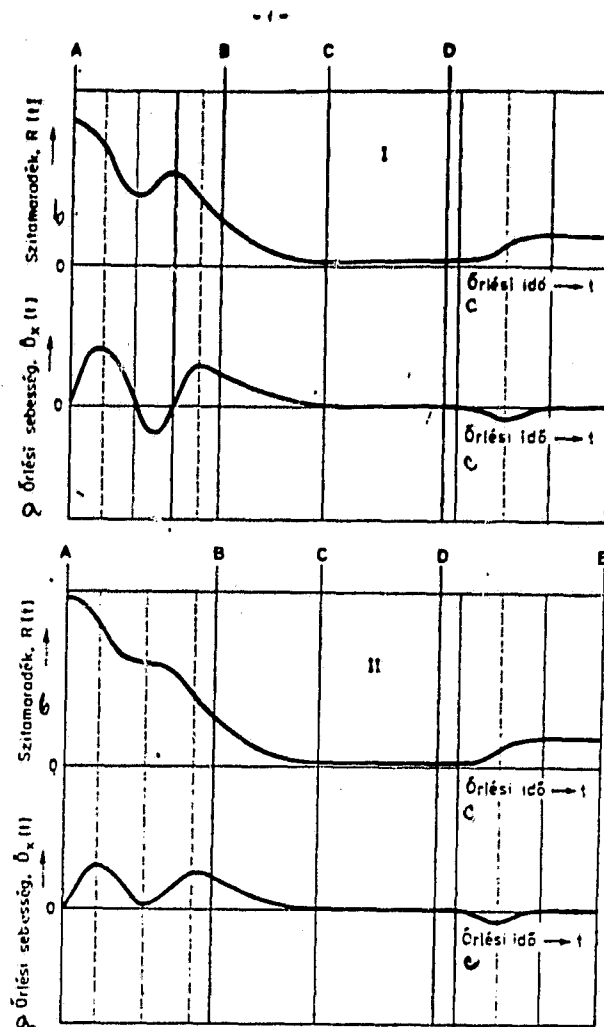


Fig. 1. Curve types characterizing the time-related changes of the sieve residue and the grinding rate.

a -- grinding rate; b -- sieve residue; c -- grinding time



Fig. 2 to 5. Presentation of the results on the surface, obtained by the two-variable function.

$R_t(x)$  sieve residue - particle size curves at a set grinding time ( $t$  - const.)

$R_x(t)$  sieve residue - grinding time curves at a set particle size ( $x$  - const.)

$D_t(x)$  grinding rate - particle size curves

$D_x(t)$  grinding rate - time curves

$D(x, t) = 0$  grinding equilibrium state curve

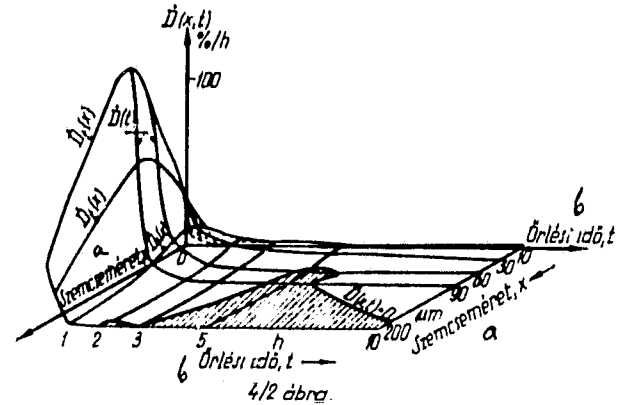
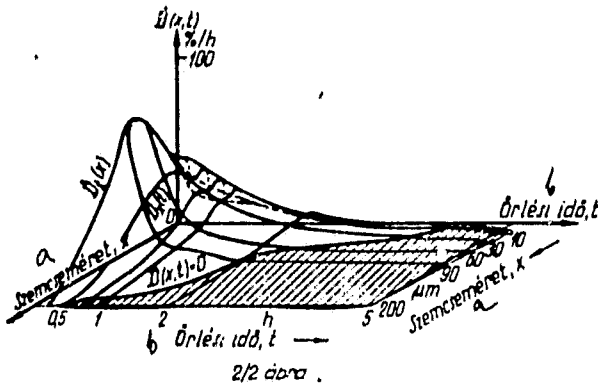
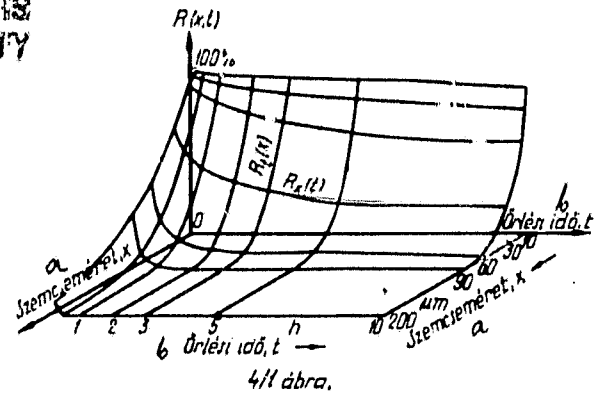
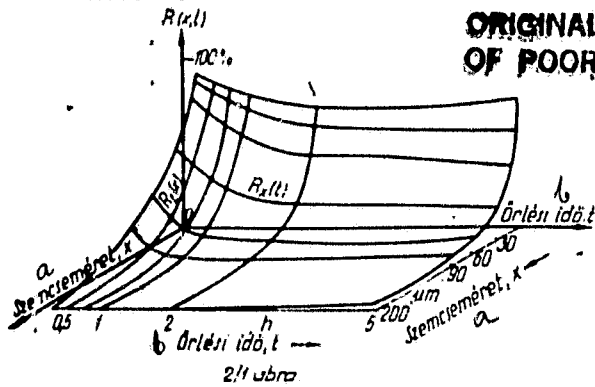
$a$  - particle size,  $b$  - grinding time

ABRA = FIGURE

Calcite **KALCIT**

Clinker (1) **KLINKER (1)**

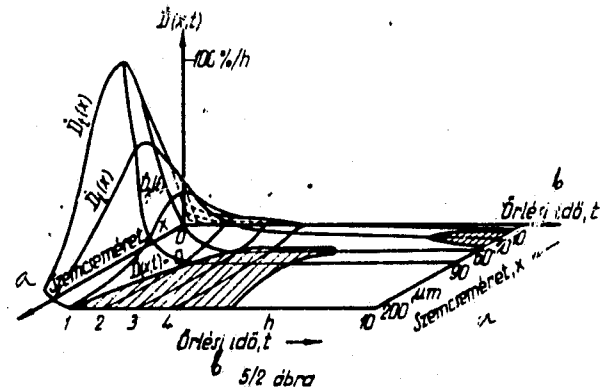
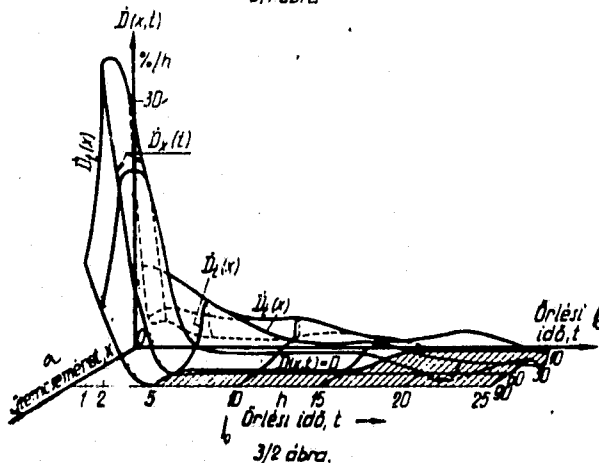
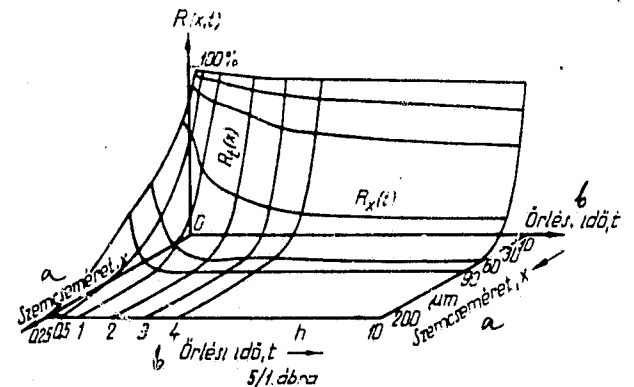
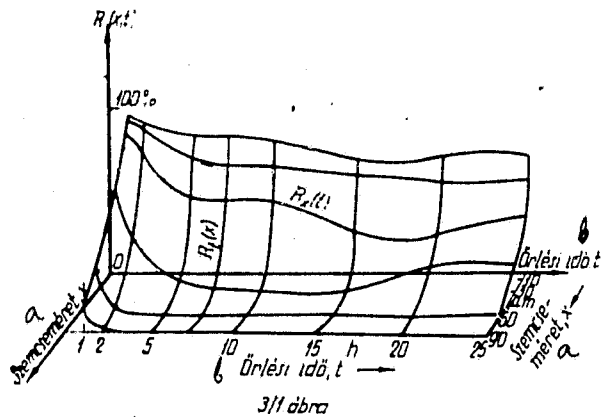
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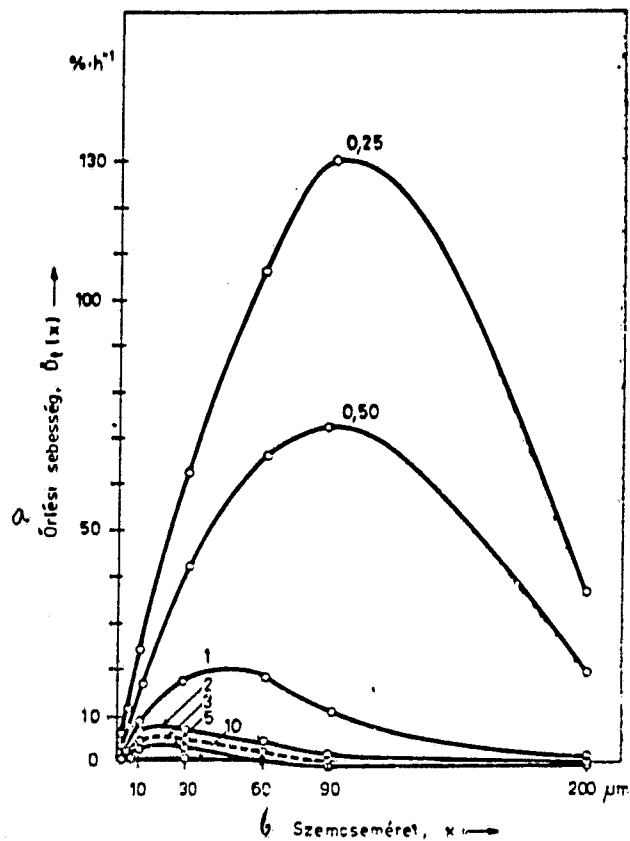
2-5. ábra. A kétváltozós függvényben nyert eredmények felületen való ábrázolása  
 $R_s(x)$  szitamaradék-szemcseméret görbék rögzített örlési idő mellett (t-állandó)  
 $D_s(x)$  örlési sebesség-szemcseméret görbék rögzített örlési idő mellett (t-állandó)  
 $D_s(t)$  örlési sebesség-idő görbék rögzített szemcseméret mellett (x-állandó)  
 $D(x,t)=0$  az örlési egyensúly állapotgörbéje

$R_s(t)$  szitamaradék-örlési idő görbék rögzített szemcseméret mellett (x-állandó)  
 $D_s(x)$  örlési sebesség-szemcseméret görbék rögzített örlési idő mellett (t-állandó)  
 $D_s(t)$  örlési sebesség-idő görbék rögzített szemcseméret mellett (x-állandó)  
 $D(x,t)=0$  az örlési egyensúly állapotgörbéje

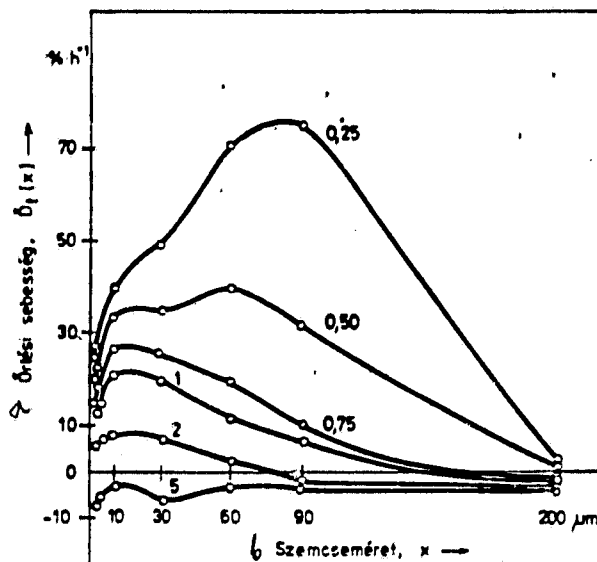
**KVARC Quartz**



Clinker (1) KLINKER (1)



Calcite KALCIT



Quartz KVARC

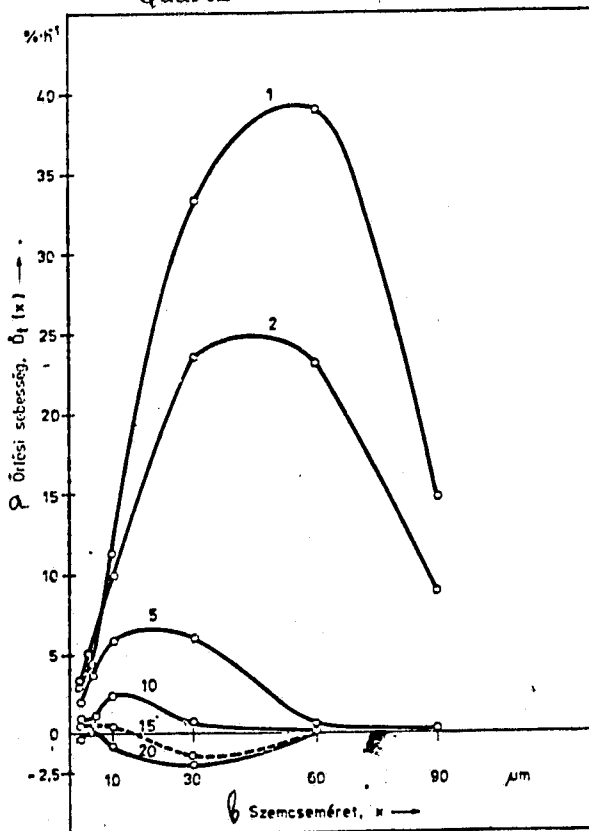


Fig. 6 to 8. Change of the grinding rate recorded at a set time as a function of the particle size (parameter: grinding time, hours)

a -- grinding rate;  
b -- particle size.

TABLE I. CERTAIN IMPORTANT CHARACTERISTICS OF EXPERIMENTAL MATERIALS

	Calcite	Quartz	Clinker (1)	Clinker (2)
Chemical composition mass %				
ignition losses				
<i>Kémiai összetétel</i> tömeg %				
Izz. vesztl.	43,98	—	0,53	0,72
SiO <sub>2</sub>	—	99,99	22,75	22,76
Al <sub>2</sub> O <sub>3</sub>	—	—	5,25	5,27
Fe <sub>2</sub> O <sub>3</sub>	—	—	2,01	3,53
TiO <sub>2</sub>	—	—	0,35	0,32
CaO	56,00	—	65,56	65,83
MgO	—	—	3,75	0,80
MnO	—	—	0,04	0,04
Na <sub>2</sub> O	—	—	0,38	0,17
K <sub>2</sub> O	—	—	0,27	0,64
SO <sub>3</sub>	—	—	nyom.	0,27
			(traces)	
Mineral composition mass %				
<i>Ásványi összetétel</i> tömeg %				
C <sub>2</sub> S (alit)			Klinker (1)	Klinker (2)
C <sub>2</sub> S (belit)			64,00* 55,89**	61,00* 53,84**
C <sub>3</sub> A			18,00 23,05	20,00 24,63
C <sub>4</sub> AF			12,00 10,51	8,00 7,99
			4,00 6,12	8,00 10,74
Density, kg.m <sup>-3</sup>				
microhardness				
MPa (N mm <sup>-2</sup> )				
Sűrűség (kg.m <sup>-3</sup> )	2690	2640	3150	3180
Mikrokeménység MPa (N mm <sup>-2</sup> )	1090	11200	—	—

\* Determined by X-ray diffractometer

\*\* Calculated according to Bogue

TABLE II. PARTICLE SIZE DISTRIBUTION OF THE EXPERIMENTAL MATERIALS  
(GRINDING PRODUCTS)

Örési idő grinding time hours	> 200 $\mu\text{m}$	> 90 $\mu\text{m}$	> 63 $\mu\text{m}$	> 30 $\mu\text{m}$	> 10 $\mu\text{m}$	> 5 $\mu\text{m}$	> 3 $\mu\text{m}$	n*	$\bar{x}$ * $\mu\text{m}$
	KALCIT Calcite								
0,25	0,15	23,45	35,25	55,72	81,40	90,09	93,72	1,0202	48
0,50	0,10	9,20	20,80	44,87	71,76	83,61	89,30	0,9381	33
0,75	0,20	6,52	14,15	37,35	63,72	78,19	86,09	0,8596	27
1,00	0,46	3,86	10,56	31,52	57,09	73,65	82,17	0,7910	23
2,00	1,20	3,90	8,50	23,23	47,44	63,92	74,22	0,6547	17
5,00**	4,20	7,01	10,05	21,25	40,92	60,95	78,03	0,5877	18
	KVARC Quartz								
1,00	0,00	12,25	33,25	61,45	86,07	93,96	96,54	1,1734	53
2,00	0,00	0,25	2,75	32,99	76,62	89,43	93,40	1,3314	25
5,00	0,00	0,00	0,00	6,34	50,30	72,84	85,31	1,2282	13
10,00	0,00	0,00	0,00	0,76	37,53	66,80	81,49	1,3753	10
15,00	0,00	0,00	0,00	0,10	27,53	61,34	70,19	1,3287	8
20,00	0,00	0,00	0,00	16,35	28,83	57,40	72,18	0,7492	11
25,00	0,00	0,00	0,00	17,06	36,87	58,60	73,74	0,7532	12
	KLINKER (1) Clinker (1)								
0,25	7,80	34,80	45,20	71,69	92,90	97,29	98,67	1,2621	83
0,50	0,70	9,70	23,90	58,58	87,78	94,99	97,48	1,2786	50
1,00	0,10	2,30	11,50	47,77	83,11	93,07	97,36	1,3356	38
2,00	0,20	1,20	6,10	39,52	76,03	90,62	96,71	1,2652	33
3,00	0,20	0,70	3,20	34,51	70,22	86,95	95,96	1,2080	29
5,00	0,20	1,00	4,00	25,77	64,04	83,45	94,17	1,1056	26
10,00	0,30	0,80	2,40	22,72	57,97	75,89	88,65	0,9548	21
	KLINKER (2) Clinker (2)								
0,25	2,10	35,20	48,20	72,65	92,62	97,92	99,12	1,2011	77
0,50	0,10	11,40	29,10	62,14	88,66	95,62	98,21	1,3768	51
1,00	0,00	1,20	8,10	46,41	82,57	93,50	97,75	1,4925	34
2,00	0,20	0,40	1,20	31,07	74,74	89,07	96,54	1,2893	29
3,00	0,60	1,00	1,30	25,45	67,86	85,53	95,02	1,1443	27
5,00	0,80	1,10	1,50	21,87	65,45	83,40	94,91	1,1090	26
10,00	0,20	0,50	0,90	17,82	56,96	76,63	92,25	1,0486	21

\* After transforming the particle size and sieve residue values, we determined the constants n and  $\bar{x}$  of the RRSB equation (n is the directional tangent of the straight line (uniformity factor), while  $\bar{x}$  is the particle size corresponding to the sieve residue of 36.8%) by means of correlation and regression calculations.

\*\* Because of strong coalescence we were not able to continue the grinding.